SANDIA NATIONAL LABORATORIES
CHEMICAL & DISPOSAL ROOM PROCESSES DEPARTMENT 6748
WASTE ISOLATION PILOT PLANT PROJECT

TOP-554

VISUAL DESCRIPTION, PETROGRAPHIC DESCRIPTION, PHOTOGRAPHY, AND SUBSAMPLING OF CLAY-SIZE MATERIALS FROM CULEBRA DOLOMITE SAMPLES

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1.0 REVISION HISTORY

None. This is an entirely new document

2.0 PURPOSE

This procedure supports clay mineral characterization studies related to the Waste Isolation Pilot Plant project and applies to the visual description , petrographic description, photography, and subsampling of clay-sized material from samples of the Culebra Dolomite Member of the Rustler Formation.

3.0 SCOPE

This procedure provides technical guidelines for both macroscopic and microscopic description of samples of Culebra Dolomite removed for clay mineral characterization studies. Requirements related to photographic documentation of samples are included. In addition, this procedure describes the required activities for the subsampling of clay-sized material from these samples.

The justification for the study is described in *Test Plan for Evaluation of Dissolved Actinide Retardation at the WIPP* (Papenguth and Behl, 1996). This TOP describes analyses done in support of *Mineralogical and Chemical Analyses of Fracture, Matrix and Vug Lining Minerals in Selected Culebra Dolomite Samples Collected Prior to 1991, Analysis Plan AP-002* (Siegel, 1996). Related TOP's and studies are described in AP-002.

4.0 SAFETY

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOPs of the laboratory where the work will be performed shall be followed.

5.0 RESPONSIBILITIES

The Principle Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure, documenting activities, and assuring that the latest revision of this document is followed.

The Quality Assurance Manager (QA Manager) is responsible for monitoring the work to assure proper implementation of this procedure and for assuring its continued effectiveness.

6.0 MACROSCOPIC VISUAL DESCRIPTIONS

Macroscopic visual descriptions of Culebra Dolomite samples will provide qualitative, descriptive information regarding the distribution of clay-sized materials in samples of the Culebra Dolomite. Descriptive information will be recorded on the Macroscopic Sample Description Form (MSDF) (Attachment A). The classification for planar and quasi-planar surfaces in the Culebra Dolomite (Attachment B) will provide the basis for visual descriptions. In addition to providing a visual description of the sample, the MSDF will serve as a summary record of additional analyses performed on the sample. The results of additional analyses will be attached to the MSDF.

The following discussion applies to the use of the MSDF (Attachment A). A unique identifier will be assigned to each sample and recorded on the MSDF. Additional information regarding the source location of the sample, the sample weight, dimensions will also be documented. A general geologic description of the sample will be included. This description may include: 1) apparent composition (e.g., mineral composition of the sample), 2) texture (crystallinity or grain size), and 3) structure (sedimentary, diagenetic, and structural features). The surfaces of the sample will also be classified using the classification scheme in Attachment B. In addition, the disposition and purpose of subsamples will be recorded. If taken, a photograph of the sample will be attached to the MSDF in the specified location. If no photograph exists, the investigator will write N/A. Information regarding additional analyses of the sample will be documented on the second page of the form and attached to the MSDF.

7.0 PHOTOGRAPHIC DOCUMENTATION

Photographs may be used to provide additional visual documentation of the occurrence of clay-sized materials in the Culebra Dolomite. This procedure applies to photographs of hand samples and photomicrographs.

It is required that a photograph log be prepared in the investigators scientific notebook (QAP 20-2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS). For photographs, this log will contain: 1) the photographer's name, 2) date, 3) roll no., 4) frame no., 5) sample identifier, and 6) notes. For photomicrographs, the following additional information must be recorded in the photograph log: 1) the microscope and ocular used, 2) the size of unique features in the photographed area, and 3) the approximate dimensions of the photographed area.

For photographs of hand samples, a photograph identification placard will be included in each photograph. The placard will display a unique sample identifier (ID). Additional optional information, including roll no, frame no., and date may be included on the placard. The placard may consist of a chalk board, a white board, or a tablet and included with the subject in each photo. A scale with either metric and English units (e.g., a GSA photo scale) will be mounted to photo ID placard.

As it is impractical to include a photographic placard in photomicrographs, no unique identifier is required to be placed in the photograph.

Once photographs are developed, a master catalog of the photographs and negatives will be prepared and maintained by the PI. The catalog will contain the negatives, ordered by roll and frame, in a separate section. Photographs will be organized by roll and frame. The roll no. and frame no. will be recorded on the back of each print. These prints will be placed in transparent sleeves and included in the master catalog. A copy of the photographic log for each roll will also be included in the master catalog.

8.0 PREPARATION OF THIN SECTIONS

Thin sections of samples for microscopic analysis or SEM or electron microprobe imaging will be prepared commercially. The disbursement of samples for thin sectioning to the commercial agent will follow QAP 13-2 (CHAIN-OF-CUSTODY). Thin sections will be prepared to standard industry specifications regarding thickness and degree of polish. Because of the presence of water-soluble minerals in samples of Culebra Dolomite, it is required that all samples be prepared using oil, not water. Thin sectioning procedures will be documented in a scientific notebook (QAP 20-2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS.

Note: Water-soluble mineral phases may be present in samples of the Culebra dolomite. All cutting, grinding, and polishing must be done under kerosene or alcohol to avoid dissolution of those phases. After all steps in which grinding grit or diamond paste is used, the sample is washed twice in isopropyl alcohol or acetone in an ultrasonic bath to remove grit and any fluids used in the cutting, grinding, or polishing process. After each polishing/impregnating step (except the final polish), samples are impregnated by covering the sample surface with impregnation material (e.g. epoxy resin) and curing in the manner described below.

Cut and trim the sample with a rock saw to form a chip or billet about 8 mm thick and up to 2.5 cm diameter. In general, chips are square or rectangular, but chip shapes may vary as required by the amount of sample available, the shape of the sample, or the friability of the samples.

Impregnate chips under vacuum with diethylene triamine and Epon 815 Resin (or equivalent), using the following procedure:

1. Place each chip in a disposable plastic beaker and fill with resin/hardener mix. Sprinkle in a few quartz grains to be used to monitor the thickness of the section during polishing.
2. Place beakers in a vacuum desiccator (heat off), close and evacuate for 5 minutes.
3. Break vacuum and wait for several minutes.
4. Close and re-evacuate for 5 minutes.

5 Break vacuum and remove samples. Cure on a preheated hot plate at 60°C for 15 minutes, or at room temperature for 24 hours.

Grind the chip surface to be mounted flat on a 220 μm grinding disc, and mount the chip of a pre-ground glass slide with Locktite 354 or 349, Buehler Epoxy Resin, or equivalent. Depending on the mounting material used, curing may take 24 hours at room temperature, 15 minutes on a 60 °C hot plate, or 10 minutes under an ultraviolet light. Cut the section to about 1 mm thickness with a trim saw, clean, re-impregnate, and grind to about 45 μm thickness on a grinder. Lap section by hand with 1000 grit abrasive on a glass plate to about 35 μm thickness using kerosene as a lubricant. Clean, impregnate, and grind the sample to about 30 μm with 9 μm diamond paste on polishing laps.

Section thickness is evaluated by optical examination of quartz or feldspar grains embedded in the epoxy. Samples should be thinned until the characteristic gray-yellow birefringence is obtained.

Once the proper thickness is attained, clean, impregnate, and polish with 1μm diamond paste until a high quality polish is obtained. A finer paste may be used for the final polish if deemed necessary. The final polish is done with a paper substrate, using kerosene as a lubricant.

9.0 PETROGRAPHIC ANALYSIS

The purpose of petrographic (microscopic) description of samples is to provide additional qualitative information regarding the distribution of clay sized materials in the Culebra Dolomite. Descriptive information will be recorded on the Thin Section Description Form (TSDF) (Attachment C). When completed, this form will be attached to the MSDF (Attachment A).

Thin section descriptions will include: 1) general composition (mineralogy) and rock type, 2) spatial variations in composition, 3) textural information, and 4) structural information (e.g., sedimentary, diagenetic, and structural features).

10.0 SAMPLING OF CLAY-SIZED MATERIAL FOR X-RAY DIFFRACTION AND BULK CHEMICAL ANALYSIS

All sample collection and subdivision will be performed in accordance with QAP 13-1 (CONDUCTING AND DOCUMENTING SAMPLE CONTROL), and sample disbursement will follow QAP 13-2 (CHAIN-OF CUSTODY). Because sample procedures for this work cannot be uniquely prescribed due to sample variability, deviations from the sampling procedures will be documented in a scientific notebook, following QAP 20-2 (PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS).

10.1 Surface Coatings of Clay-Sized Material

The weight of clay-sized material per surface area sampled should be estimated for the surface features of each sample as follows:

1. A sketch of the geometry of the surfaces covered or partially covered with clay-sized material should be made on the MSDF.
2. All of the clay-sized material should be carefully removed (e.g. scraped or ground off). Care should be taken to minimize removal of the underlying matrix.
3. The scraped material should be weighed, per requirements of TOP 556, and the weight entered onto the MSDF.
4. Next, the scraped material should be ground and mixed until its appearance is homogeneous in color and particle size as judged by visual inspection with the unaided eye.
5. Subsamples of this ground homogeneous material should be taken and weighed for bulk chemical, x-ray diffraction and other analyses.
6. The subsamples should be assigned uniques identifiers; the ID's and weights of the subsamples should be recorded in the MSDF.

10.2 Subsamples of the bulk (matrix) rock

A subsample of the matrix underlying the surface sampled in Section 9.1 above should be taken as follow:

1. The scraped surface should be washed with deionized water to remove residual particles adhering to the surface as determined by visual inspection.
2. A sample of the matrix should then be broken off, weighed, per requirements of TOP 556, and then ground to a homogeneous powder.
3. Subsamples of this ground homogeneous material should be taken and weighed for bulk chemical, bulk x-ray diffraction, clay extraction and other analyses.
4. The subsamples should be assigned uniques identifiers; the ID's and weights of the subsamples should be recorded in the MSDF.

11.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with QAP 5.3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program. The following discussion applies to the manufacture of thin sections.

11.1 STANDARDS

The quality of a polished thin section is dependent upon two variables; the quality of the surface polish, and the thickness of the section. The quality of the surface polish is monitored and subjectively determined to be acceptable by the thin section technician during production of the section. The thickness of the section is determined by examination of the optical properties of minerals within the section; the section is of the proper thickness (~30 microns) when quartz or feldspar grains in the section exhibit characteristic gray or yellow-gray birefringence. As the Culebra Dolomite does not contain sufficient quantities of these phases, a few quartz grains will be sprinkled into the epoxy surrounding the sample during impregnation. The thickness of these grains will be monitored during polishing to ensure that the proper thin section thickness is achieved.

11.2 PERFORMANCE TEST CRITERIA

Each thin section will be examined for surface scratches and proper thickness prior to use.

11.3 CORRECTIVE ACTION

In the event that a thin section is found unacceptable (it is heavily scratched or of improper thickness), it will be returned to the thin section technician for further polishing. If this cannot be achieved without overthinning the section, it will be discarded and a new section will be made from the original billet. All corrective actions will be recorded in laboratory notebooks, and will follow the procedures described in QAP 16-2, CONDITIONS ADVERSE TO QUALITY AND CORRECTIVE ACTION (Revision 1. 7/31/95).

All entries into laboratory notebooks will conform to the requirements of QAP 20.2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date 7/31/95).

11.4 CALIBRATION

This procedure requires no calibrations.

12.0 QA RECORDS

The following QA records shall be prepared and submitted to the Sandia WIPP Central File, per identified QAPs:

1) Macroscopic sample description form (MSDF) - QAP 20-2

2) Scientific notebook - QAP 20-2
3) Photograph log and master catalog (negatives and photographs) -QAP 20-2
4) Thin section description forms (TSDF) - QAP 20-2
5) Chain of Custody -QAP 13-2

Note: 2 copies of photographs and the photograph catalog will be submitted.

13.0 FORMS

A Chain-of-Custody form (Attachment D) will accompany each rock sample for which a thin section is to be prepared, and will be returned to the Project Scientist, along with the prepared section and the thin section billet, upon completion of the work. The chain of custody for each sample will be maintained as described in QAP 13-2, CHAIN OF CUSTODY (Revision 1, 7/31/95).

14.0 REFERENCES

Papenguth, H. W. and Behl, Y.K., 1996, Test Plan for Evaluation of Dissolved Actinide Retardation at the Waste Isolation Pilot Plant, Test Plan 96-02, Rev 0, Sandia National Laboratories, Waste Isolation Pilot Plant Project document.

QAP 13-1, CONDUCTING AND DOCUMENTING SAMPLE CONTROL (Revision 1, effective date: 7/31/95)

QAP 13-2, CHAIN-OF-CUSTODY, (Revision 1, effective date: 7/31/95)

QAP 16-2, CORRECTIVE ACTION, (Revision 1, effective date: 7/31/95)

QAP 20-2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date: 7/31/95)

Siegel, M. D., 1996, Mineralogical and Chemical Analyses of Fracture, Matrix and Vug Lining Minerals in Selected Culebra Dolomite Samples Collected Prior to 1991, Analysis Plan AP-002, Rev. 0, Sandia National Laboratories, Waste Isolation Pilot Plant Project document.

ATTACHMENT A
MACROSCOPIC SAMPLE DESCRIPTION FORM

Sample # _____

Borehole: _____ Initial sample weight: _____

Depth. _____ Approx. Dimensions (mm): _____

Sample Source:

Collected by: _____ Date: _____

For the following purpose: _____

_____

_____

Description. Include a general sample description plus a detailed description of the appearance and geometry of the suface to be sampled. Include a sketch of the surface to be sampled.

Classification of surface to be sampled: _____

Sub-sample	Sub-sample ID	Sample weight, g
Coating material collected		
1) XRD		
2) Bulk chemical analysis		
Thin section chip		
SEM chip		
Matrix sample collected		
1) XRD		
2) Bulk chemical analysis		
Other:		
Other:		

Photograph of Sample #:_____

Detailed Analysis Checklist

XRD Results

Was the bulk fracture coating material analyzed by XRD? _____

Was the material treated to remove dolomite or gypsum (e.g. EDTA dissolution) and re-examined?_____

Was the material glycolated and re-examined?_____

If yes to any of these, attach a detailed description of the phases present, the basis for mineralogical identification. an estimate of the relative abundance of the phase, and a copy of the XRD patterns.

Bulk Chemical analysis

Was a bulk chemical analysis made of the fracture-coating material? _____

If yes, attach a summary of the results.

Thin sections

Was a thin section made?_____ If yes, attach a detailed description and photographs.

SEM or Microprobe imaging

Was the sample examined by SEM or Microprobe?_____ If yes, attach a detailed description and photographs.

ATTACHMENT B
CLASSIFICATION SCHEME FOR PLANAR SURFACES IN THE CULEBRA DOLOMITE

Classification Scheme for Planar and Quasi-planar Features in the Culebra Dolomite

Feature

Coating	Cross-cutting fracture	Bedding plane fracture	Parting	Seam
Dark gray or brown materia ("clay")	A1	B1	C1	D1
Orange/yellow material ("Fe oxyhydroxides")	A2	B2	C2	D2
Selenite	A3	B3	C3	D3
Powdery dolomite	A4	B4	C4	D4

Cross-cutting fracture — a fracture which cuts across bedding features.
Bedding plane fracture — a fracture parallel or sub-parallel to bedding (usually occurring through vug-rich zones); this class does not include partings.
Parting — a bedding plane fracture following compositional layering, usually a thin (<1mm) "clay"-rich layer.
Seam — a "clay" layer >1mm tl

Note: Surfaces which have mixtures of the coatings will be designated with combinations numbers (ie. A12, A34, etc.), corresponding to the coatings listed in the chart in orde increasing abundance.

ATTACHMENT C
THIN SECTION DESCRIPTION FORM

THIN SECTION DESCRIPTION FORM

SAMPLE ID:	DATE:	INVESTIGATOR

PURPOSE:

DESCRIPTION
(Additional sheets may be attached)

Photomicrographs: Yes_____, No_____.

Roll No._____

Frame No._____

ATTACHMENT D
CHAIN OF CUSTODY FORM

APPENDIX A

WASTE ISOLATION PILOT PLANT
Sandia National Laboratories

Chain-of-Custody

Form Number: 401 Effective: 7/31/95

Procedure: QAP 13-2
Revision: 1
Page _____ of _____

ORIGINATOR'S SIGNATURE: _____ ORIGINATOR'S PRINTED NAME: _____ CONTROL NO.: _____

ORGANIZATION NO.: _____ TIME: _____ DATE: _____

SAMPLE COLLECTION INFORMATION (only)

DATE: _____ LOCATION: _____ SAMPLER'S NAMES _____

TIME: _____ PROCEDURE NO.: _____

ASSOCIATE TEST: _____

SAMPLE DESCRIPTION

SPECIAL INSTRUCTIONS: _____

METHOD OF TRANSPORT: _____

SAMPLES SHIPPED TO: _____

NOTE: Samples or materials that are similar in type or matrix may be entered on the same form. Use Form 401 continuation page if more space is required.

Sample or Material ID Number	Type of Item	Description of Material (see note above)	Amount of Material	Comments (conditions, instructions, etc.)

CUSTODY TRANSFER: Name / Organization / Date / Time

Relinquished by: _____ Received by: _____ Name / Organization / Date / Time

Relinquished by: _____ Received by: _____

Relinquished by: _____ Received by: _____

Final Recipient: Note conditions and sign "Received by." Maintain a copy of the completed form with the samples, and send the original to: SNL WIPP Central Files (SWCF), PO Box 1957, Carlsbad, NM 88220.

Page 1 of 2 page form

SWCF File Code: _____

WBS# _____

1.1 GENERAL

This section establishes requirements for creating and maintaining an organizational structure to implement the Quality Assurance Program for the Civilian Radioactive Waste Management Program. This section also provides a description of the OCRWM organization and other affected organizations.

1.2 REQUIREMENTS

Each affected organization shall prepare controlled documents, accepted by the responsible organization with immediate authority over the affected organization (next-higher-level organization), that describe internal and external organizational interfaces, organizational structures, requirements, and responsibilities for its scope of work.

1.2.1 Line Management

Each affected organization shall identify the responsibilities and authorities of those organizations and management positions responsible for achieving and maintaining quality.

1.2.2 Quality Assurance Management

Each affected organization shall identify the management position within the organization responsible for performing quality assurance functions. This position shall be occupied by an individual with appropriate knowledge and experience in management and quality assurance. The position shall:

A. Be at the same or higher organization level as the highest line manager directly responsible for performing work subject to QARD requirements.

B. Be sufficiently independent from cost and schedule considerations.

C. Have the organizational freedom to effectively communicate with other senior management positions.

D. Be responsible for interpreting and approving quality assurance program requirements as they apply to the affected organization's scope of work. ✓

E. Have no other assigned responsibilities unrelated to the quality assurance program that would prevent full attention to quality assurance matters.

F. Be responsible for identifying quality problems, initiating, recommending, or providing solutions to quality problems, and verifying solutions to quality problems.